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PRIMARY STUDY OF CELLULOSE ACETATE HOLLOW FIBER AS A GREEN MEMBRANE APPLIED TO HEMODIALYSIS

Yanuardi Raharjo, Siti Wafiroh, Mahdya Nayla, Vita Yuliana, Mochamad Zakki Fahmi

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ABSTRACT

Hollow fiber membranes are widely applied in the health field, especially for hemodialysis. The purpose of this study was to determine the effect of the coagulant bath temperature and the air gap on the mechanical properties of cellulose acetate hollow fiber membranes prepared for hemodialysis application. The membranes prepared contained 22 % of cellulose acetate, 51 % of acetone, and 27 % of formamide. They were printed by the phase inversion method at five coagulant bath temperature values of 5ºC, 10ºC, 15ºC, 20ºC, and 25ºC with air gap distance of 10 cm, 15 cm, 20 cm, 25 cm, and 30 cm. The membranes tensile test as well as their performance determined by flux, rejection and morphology were carried out by SEM. The average pore size obtained referred to 45 nm. The optimum values were provided in the course of coagulation at a bath temperature of 5ºC and an air gap distance of 25 cm. They referred to thickness of 0.28 mm, stress of 2,879.58 N/m^2, strain of 0.179, Young modulus of 14617.16 N/m^2, urea flux of 49.4 L/m^{2}h and rejection of 19.65 %.

Keywords: cellulose acetate, hollow fiber membrane, hemodialysis.

INTRODUCTION

Membranes are generally defined as semi-permeable thin layers that function as a specific filter separating the components of a mixture [1]. The separation technology using membranes is currently growing very rapidly. This is determined by a number of advantages [2]: (i) it requires a small amount of energy, which excludes molecular structure alteration of the substance separated; (ii) it can be performed at a room temperature; (iii) it does not require additional chemical substances during the separation process.

Membrane application is currently widely used for medical purposes, especially in hemodialysis. There it saves lives of patients requiring renal replacement therapy [3] and it is worth noting that the number of such patients in Indonesia is approximately equal to 150 000 [4].

Hemodialysis is a medical renal replacement therapy required because of decreased acute or chronic kidney function [5]. Hemodialysis is in fact connected with removal of toxic metabolism particles in the artificial kidney. The process takes place in a dialysis machine, where the blood of patients with a kidney failure discharged through their veins is cleansed through the process of diffusion and ultrafiltration with dialysate [6]. The substances filtered out in the course of hemodialysis refer mainly to water, salts, ammonia, creatinine and urea. The dialysate solution used contains chemicals similar to those found in the body, such as sodium, potassium, calcium, magnesium, chloride, bicarbonate, acetate, acetic acid and glucose. The process results in red blood cells increase proceeding at a maintained thermal equilibrium. Then the blood gets an access to the blood circulation in the patient's body [7].

Hemodialysis membranes use predominantly hollow fiber membranes. They are preferred when compared to flat membrane because the latter frequent fouling. Besides the flat membranes have also a relatively small surface area. Furthermore, the hollow fiber membranes can provide better performance [8] due to the larger flat surface area.

This study uses cellulose acetate as a material for hollow fiber membranes preparation. The aim is to use them to filter out urea. The choice of cellulose acetate is determined by the material tolerance [9] in respect to human blood temperature of 37ºC and pH ranging from 7.35 to 7.45. Besides, cellulose acetate is self-degrading, which is in accord with green chemistry principles.

EXPERIMENTAL

Acetone, formamide, cellulose acetate, sodium azide, distilled water, ice cubes, urease, phenolphtalein indicator, creatinine and urea were used. All materials were of a (p.a.) quality. The laboratory equipment was of a conventional type. The analytical equipment employed referred to UV-Vis Double Beam Shimadzu, autograph AG-10 TE Shimadzu and SEM (Scanning Electron Microscope) ZEISS EVO MA 10.

Preparation of hollow fibers

The composition used for preparation of hollow fibers referred to 22 % of cellulose acetate, 27 % of formamide and 51 % of acetone. The amount of cellulose acetate required was dissolved in acetone. Then formamide was added under constant stirring for ca 2 h using a magnetic stirrer. The homogeneous solution obtained, called a dope solution, was kept overnight to remove the air bubbles trapped during the dissolution process.

Then the dope solution was poured into the first and second tube of the hollow fiber printing equipment. The first tube was connected with a nitrogen tube. Nitrogen was used to apply pressure on the dope solution, so that it could flow and let the trapped air out. A flow meter was used. The distances between spinnerets and cold water bath tub was about 10 cm, furthermore the temperature was arranged 5°C, 10°C, 15°C, 20°C and 25°C. Those procedures were repeated varying the air gap distance to 15 cm, 20 cm, 25 cm, 30 cm. The membranes printed were stored for 1 day in cold water and then washed with running water to remove any remaining solvent. The membranes were subsequently cut and stored in a solution of 1 % sodium azide.

Thickness determination

The thickness of the cellulose acetate hollow fiber membranes was obtained by a conventional procedure using a micrometer screw. Precision of 0.01 mm was achieved. The average values of those obtained were further used.

Mechanical properties determination

A tensile test of the membranes prepared was carried out. Loads of the order of kN were applied, while the membranes were pulled with a speed of 1 cm/ min. The force (kN) required to break the membrane and the accompanying change in length (Δl) were recorded by the tensile test equipment. The values obtained provided the estimation of the stress, the strain, and the Young modulus.

Determination of the hollow fiber membrane performance

Determination of permeability (flux) was done in a cross flow filtration cell. The water flux value was determined on the ground of the volume of distilled water collected every 20 min in the course of a certain time interval. At a later stage the water was replaced by a solution of urea. Its flux value was determined at intervals of 60 min.

The membrane selectivity was determined by the value of the rejection coefficient (R). Its value estimation required the measurement of the feeding solution concentration before (C_{before}) and after (C_{after}) passing through the membrane. The calculation was done using the following formula:

$$R(\%) = 1 - \frac{C_{after}}{C_{before}} \times 100\%$$

The volume of the feeding solution was accommodated in a separate container. The samples used for the determination were of a volume of 15 ml. The levels of urea in the initial sample and that permeating through the membrane were determined spectrophotometrically. The solution of urea used was generally compatible in respect to its concentration in human blood. It was 0,05 M. The volumes of the feeding urea solution and that pass through the membrane were separately contained. Samples of 15 mL were used for the quantitative spectrophotometric evaluation.

Determination of the hollow fiber membrane morphology

The membrane morphology was determined by SEM. The membrane samples had to be dried.

RESULTS AND DISCUSSION

Results referring to the preparation of cellulose acetate hollow fiber membrane

Various compositions of the dope solution were obtained. The optimum composition of the dope solution referred to 22 % of cellulose acetate, 51 % of acetone and 27 % of formamide. A water flow rate of 150 mL/min was used, while the nitrogen gas provided pressure of 5 kg/cm^2.

Thickness measurement results

The hollow fiber membranes were cut longitudinally and then unfolded prior to thickness measurement. An air gap distance of 10 cm produces membranes of the highest thickness. It is so because the dope solution reaches quickly the coagulation bath decreasing the contact time with the air. As the air contact time affects the evaporation of the solvent, the shorter air gap distance brings about a thicker membrane formation. At a distance of 20 cm, the thickness decreases, but it is greater than that obtained in at an air gap distance of 15 cm. The investigation of Wang et al. [12] shows that the outer diameter of the hollow fiber membrane increases with increase of the air gap distance from 5 cm to 30 cm, while the tendency of the inner diameter change remains unclear. At an air gap of 20 cm the internal diameter of the hollow fiber membrane found in this study is smaller than that obtained at an air gap distance of 15 cm. This shows that the membrane thus obtained is thicker than that produced at an air gap distance of 15 cm.

The hollow fiber membrane thickness decreases with increase of the air gap distance ranging from 20 cm to 30 cm. This is expected because of the effect of the contact time with air on the diffusion rate in the phase inversion process. But it is worth adding that the elongation of membrane voltage during the printing process affects also the force of gravity [11].

Results referring to the mechanical properties of the hollow fiber membranes

The increase of the coagulant bath temperature decreases the voltage. This occurs because the solvent diffusion process is slower at lower temperatures. Thus denser membranes are obtained. But the dense membrane pore structure results in greater membrane tension value [13]. Hence, the decrease of the solvent diffusion rate increases not only the density, but the membranes voltage as well.

Acetone evaporates and is replaced by water in the tub coagulant. So the higher the temperature, the faster is the diffusion process. This in turn increases the pore volume and the strain [13]. However, the strain observed at 20ºC is lower than that found at 15ºC. This is most probably due to the fact the printing is manual. The specific strain found at 25ºC is much higher than that obtained at a temperature of 20°C. This is so because the rate of acetone diffusion increases significantly. Cellulose acetate hollow fiber membranes of an optimal strain value are printed at a coagulant bath temperature of 5°C - the strain is the lowest under these conditions.

The tensile strength at break (stress) increases with increase of the air gap distance. The nature is brittle at an air gap distance of 10 cm as the amount of stress to deform the material or material's ability to resist a deformation is small. If the outer deformation increases, then the plastic limit will be exceeded and the material will become disconnected. A hollow fiber membrane produced at an air gap distance of 25 cm has the greatest tensile strength. That is because the molecules within the membrane grow denser, which in turn increases the tensile strength [15]. The air gap distance affects the stress strength of hollow fiber membranes. The higher membrane stress and strength result in greater strength to deform it. This is due to the fact that the increase of the air gap distance leads to decrease of the diameter of membrane and hence to increase of its density [11]. The latter effect decreases the size of the pores. Thus it can be concluded that the stress strength increases with the increase of the air gap distance. The stress of hollow fiber membrane produced at an air gap distance of 20 cm is less than that of 30 cm. These results can be attributed to the thickness of the membrane, which is greater at an air gap distance of 20 cm than that at an air gap distance of 15 cm. Besides, the membrane surface area is also larger, while the force required to break the membrane decreases with the air gap distance increase from of 15 cm to 20 cm as the stress produced is smaller.

Furthermore, the hollow fiber membranes stretch tends to increase with the air gap distance increase. The increase of the strain results in decrease of the size of the membrane pores at deformation. The great strain in case of an air gap distance of 25 cm is attributed to stronger bonds between the molecules.

Young's modulus is a measure of stiffness of a material. A greater Young's modulus value refers to a smaller elastic strain of the material, i.e. to a more rigid material. These properties are of importance in maintaining the size of the membrane pores under greater pressure application. The formula to calculate the Young's modulus is based on the ratio of the voltage and the stretch. The overall mechanical test data show that the optimal stress value is 2,879.58 N/m^2, that of stress is 0,197, while the Young's modulus has a value of 14,617.16 N/m^2.

Results referring to hollow fiber membrane performance

The urea and creatinine feed solutions used were separated by cellulose acetate hollow fiber membranes in a cross flow using gas pressure to push the feed solution out of the membrane. Flux value followed at intervals of 60 min was calculated by dividing the volume permeating through the cross-sectional area of the membrane by the separation time. The concentration of the solution was determined spectrophotometrically using a corresponding standard curve. The linear regression equation y = 0,89x + 0.714 was used. The flux value obtained in this study refers to 49.4 L/m^2h, while the rejection amounts to 19.65 % or clearance to 80.35%. The high flux value indicates minimization of fouling occurrence, while the low rejection value is a measure of the high capability of the hollow fiber membrane to pass creatinine and urea.

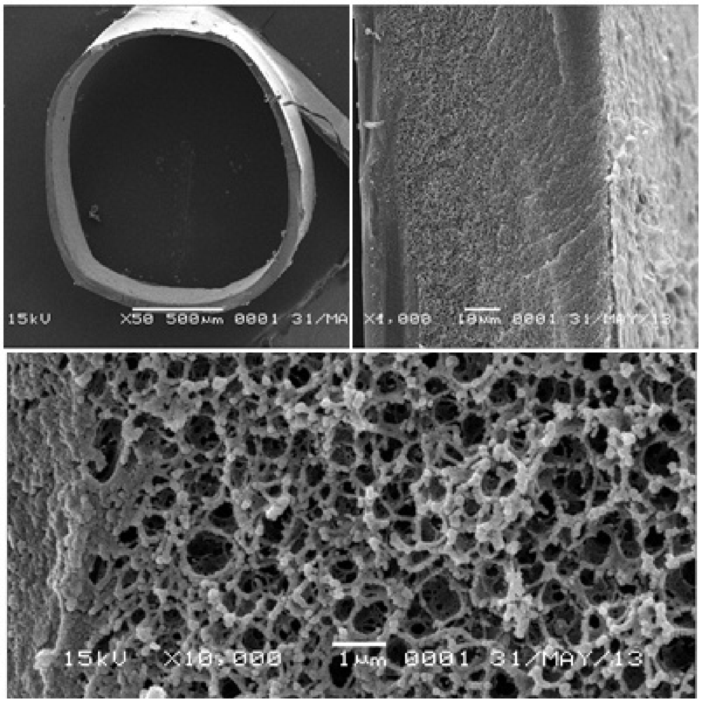

Fig. 1. Cross section of hollow fiber cellulose acetate membrane.

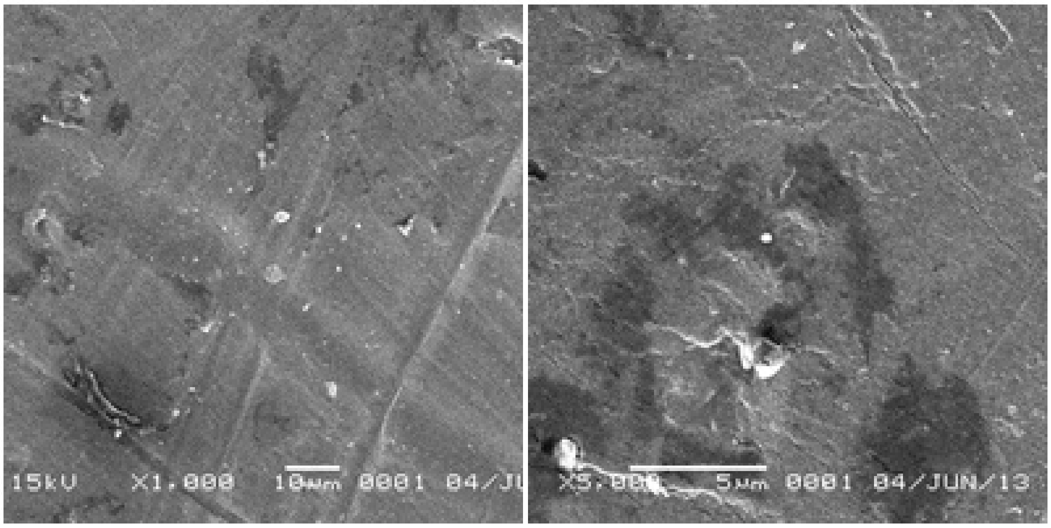

Fig. 2. Cellulose acetate hollow fiber membrane surface.

Results referring to hollow fiber membrane morphology

The SEM analysis carried provides information referring to the membranes cross-section (Fig. 1) and surface (Fig. 2).

It is found that the membrane piores size is equal to 0.45 μm. As the creatinin molecule has a radius of 30 nm [16], it is evident that its size is smaller than that of the membranes pores. Hence, creatinin can pass through the membrane.

CONCLUSIONS

Coagulation bath temperature and air gap affects the mechanical properties of the hollow fiber membrane of cellulose acetate membrane thickness, stress, strain, and Young modulus. The slower rate of diffusion at low temperatures determines regular and uniform pore formation which in turn provides optimum mechanical properties. The optimum temperature of the coagulant bath refers to 5ºC. The smallest pore diameters are achieved at an air gap distance of 25 cm. The hollow fiber membrane of cellulose acetate obtained at a coagulation bath temperature of 5ºC and air gap distance of 25 cm has the following optimal characteristics: thickness of 0.28 mm, stress of 2,879.58 N/m^2, strain of 0.179, Young modulus of 14,617.16 N/m^2, a flux of 49.4 L/m^{2}h, and a coefficient of rejection creatinine and urea rejection of 19.65 %.

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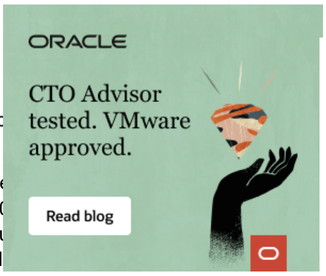

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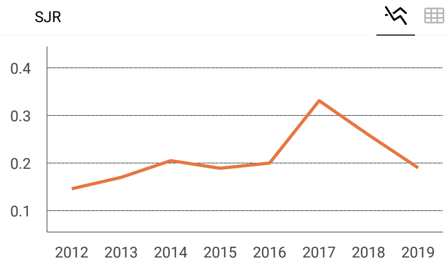

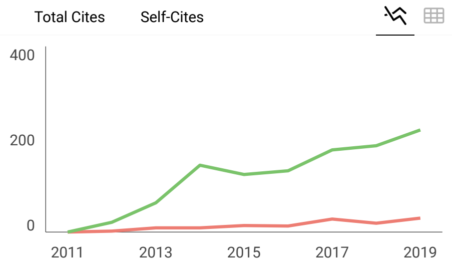

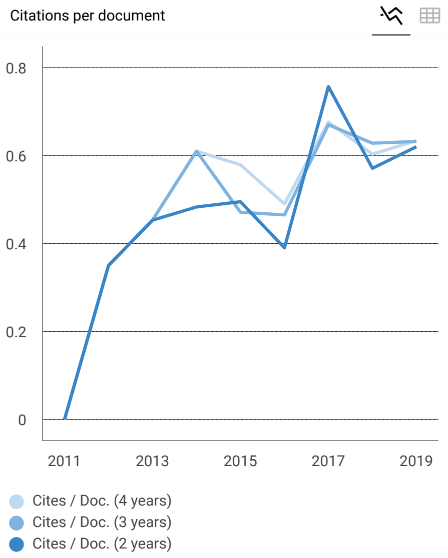

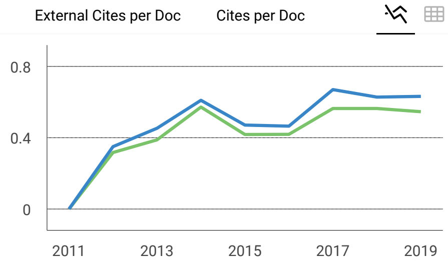

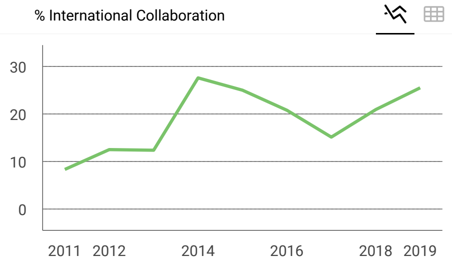

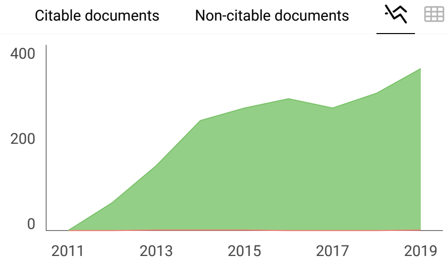

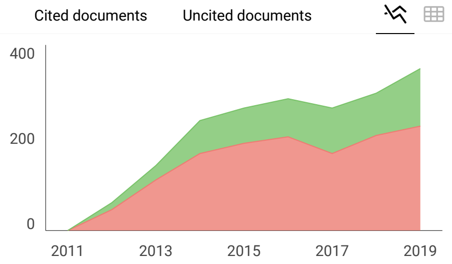

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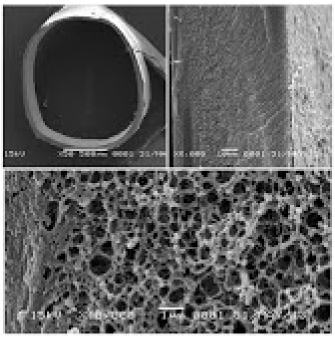

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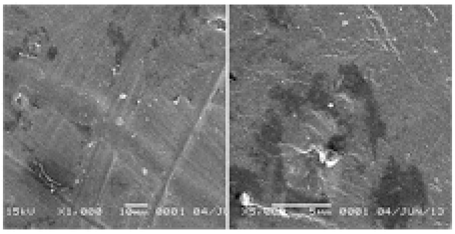

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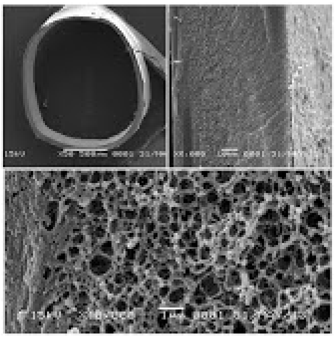

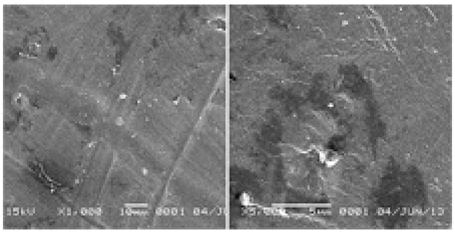

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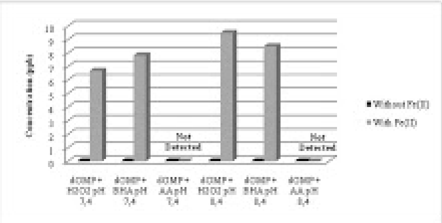

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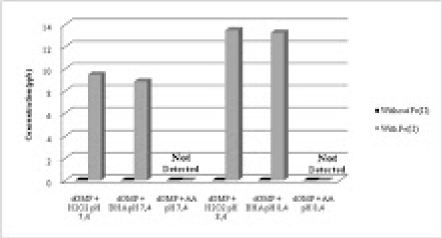

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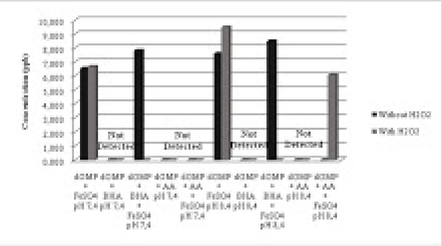

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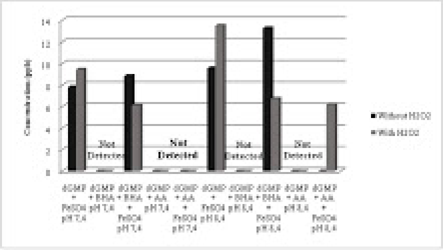

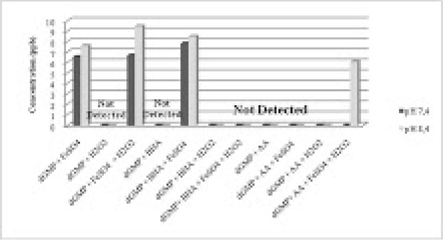

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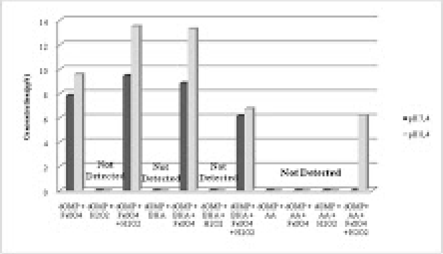

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International Conference

"Collaboration Seminar of Chemistry and Industry (CoSCI-2016)"

5-6 October, 2016

Surabaya, Indonesia

Invited Editor

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